

Certificate

Standard Reference Material 4361C Hydrogen-3 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive hydrogen-3, as water, in 500 mL of distilled water. The solution is contained in a 500-mL borosilicate-glass bottle. The SRM is intended for the calibration of beta-particle counting instruments and for the monitoring of radiochemical procedures.

Radiological Hazard: The SRM bottle contains hydrogen-3 with a total activity of approximately 1100 Bq. Hydrogen-3 decays by beta-particle emission. None of the beta particles escape from the SRM bottle. During the decay process no photons are emitted. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]*. There is no detectable external radiation. The SRM should be used only by persons qualified to handle radioactive material.

Chemical Hazard: The SRM bottle contains only distilled water. There is no chemical hazard.

Storage and Handling: The SRM should be stored and used at a temperature between 5 and 35 °C. The solution in an unopened bottle should remain stable and homogeneous until at least September 2008. The bottle (or any subsequent container) should always be clearly marked as containing radioactive material. If the bottle is transported it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations.

Preparation: This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, L.R. Karam, Group Leader. The overall technical direction and physical measurements leading to certification were provided by L.L. Lucas and M.P. Unterweger of the Radioactivity Group. The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program.

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Gaithersburg, Maryland 20899 June 1999 Half-life and text revised October 2000 Text revised and expiration date extended September 2004 Robert L. Watters, Jr., Chief Measurement Services Division This page intentionally left blank.

PROPERTIES OF SRM 4361C

Certified values

| Radionuclide | Hydrogen-3 |
|--------------------------------------|--|
| Reference time | 1200 EST, 03 September 1998 |
| Massic activity of the solution [b]* | 2.009 Bq·g ⁻¹ |
| Relative expanded uncertainty (k=2) | 0.76% [c] [d] |
| Solution density | $(0.998 \pm 0.002) \text{ g·mL}^{-1} \text{ at } 20 \text{ °C } [e]$ |

Uncertified values

| Physical Properties: | | | | | |
|--|---|--------------------------------------|------------------------------------|--|--|
| Source description | Liquid in a 500-mL borosilicate-glass media bottle with a teflon-lined screw cap | | | | |
| Solution mass | Approximately 500 g | | | | |
| Chemical Properties: | | | | | |
| Solution composition | Chemical Formula | Concentration (mol·L ⁻¹) | Mass Fraction (g·g ⁻¹) | | |
| | H ₂ O ³ HHO | 55 2×10^{-12} | $1.00 \\ 4 \times 10^{-14}$ | | |
| Radiological Properties: | | | | | |
| Radionuclidic impurities | None detected [f] | | | | |
| Half lives used | Hydrogen-3: $(4500 \pm 8) d [g] [5]$ | | | | |
| Calibration method and measuring instrument(s) | $4\pi\beta$ gas counting of SRM 4927E using the NIST length-compensated internal gas proportional counters and intercomparison of SRMs 4927E / 4926E / 4361C using two $4\pi\beta$ liquid-scintillation counting systems. [h] | | | | |

^{*}Notes and references are on pages 5 and 6.

EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [c] [d]*

| Input Quantity x_i , the source of uncertainty (and individual uncertainty components where appropriate) | Method Used To Evaluate $u(x_i)$, the standard uncertainty of x_i (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods | Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$, (%) [i] | Relative Sensitivity Factor, $\left \frac{\partial y}{\partial x_i} \right $. (x_i/y) [j] | Relative Uncertainty Of Output Quantity, $u_i(y)/y$, $(\%)$ [k] |
|---|--|--|---|--|
| Massic count rate of SRM 4927E, corrected for background and decay [h] | Standard deviation of the mean for 23 sets of gas counting measurements (A) | 0.18 | 1.0 | 0.18 |
| Gram-mole measurements | Estimated (B) | 0.20 | 1.0 | 0.20 |
| Live time [p] | Estimated (B) | 0.10 | 1.0 | 0.10 |
| Extrapolation of count-rate-versus-energy to zero energy | Estimated (B) | 0.20 | 1.0 | 0.20 |
| Half life of H-3 | Standard uncertainty of the half life (A) | 0.18 [m] | 0.009 [n] | 0.002 |
| Liquid-scintillation intercomparison of SRM 4926E and SRM 4927E | Standard deviation of the mean for 7 sets of liquid-scintillation measurements (A) | 0.06 | 1.0 | 0.06 |
| Dilution of SRM 4926E to make SRM 4361C | Estimated (B) | 0.12 | 1.0 | 0.12 |
| Radionuclidic impurities | Limit of detection (B) [q] | 100. | 0.0005 | 0.05 |
| Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$, (%) | | | | |
| Coverage Factor, k | | | | <u>x 2</u> |
| Relative Expanded Uncertainty of the Output Quantity, U/y, (%) | | | | 0.76 |

NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One μSv is equal to 0.1 mrem. Distance from Bottle (cm): 1 30 100 Approximate Dose Rate ($\mu Sv/h$): <0.1 (Not detectable)
- [b] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [c] The reported value, y, of massic activity (activity per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as $y = f(x_1, x_2, x_3, ... x_n)$, where f is a mathematical function derived from the assumed model of the measurement process. The value, x_i , used for each input quantity i has a **standard uncertainty**, $u(x_i)$, that generates a corresponding uncertainty in y, $u_i(y) = |\partial y/\partial x_i| \cdot u(x_i)$, called a **component of combined standard uncertainty** of y. The **combined standard uncertainty** of y, $u_c(y)$, is the positive square root of the sum of the squares of the components of combined standard uncertainty. The combined standard uncertainty is multiplied by a **coverage factor** of k = 2 to obtain U, the **expanded uncertainty** of y.

Since it can be assumed that the possible estimated values of the massic activity are approximately normally distributed with approximate standard deviation $u_c(y)$, the unknown value of the massic activity is believed to lie in the interval $y \pm U$ with a level of confidence of approximately 95 percent.

For further information on the expression of uncertainties, see references [2] and [3].

- [d] The value of each component of combined standard uncertainty, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval U/2 to 2U (i.e., within a factor of 2 of the estimated value).
- [e] The stated uncertainty is two times the standard uncertainty.
- [f] The estimated limit of detection for radionuclidic impurities is 0.001 Bq·g⁻¹.
- [g] The stated uncertainty is the standard uncertainty.
- [h] Extensive gas-counting measurements were made on the SRM 4927E solution during 1998 and 1999. The SRM 4926E solution was intercompared with the SRM 4927E solution using liquid-scintillation counting. The SRM 4926E solution was then quantitatively diluted, using distilled water with a measured hydrogen-3 content, to make the SRM 4361C solution. The 4926E / 4361C massic activity ratio was confirmed by liquid-scintillation counting.
- [i] Relative standard uncertainty of the input quantity x_i .
- [j] The relative change in the output quantity y divided by the relative change in the input quantity x_i . If $|\partial y/\partial x_i| \cdot (x_i/y) = 1.0$, then a 1% change in x_i results in a 1% change in y. If $|\partial y/\partial x_i| \cdot (x_i/y) = 0.05$, then a 1% change in x_i results in a 0.05% change in y.

- [k] Relative component of combined standard uncertainty of output quantity y, rounded to two significant figures or less. The relative component of combined standard uncertainty of y is given by $u_i(y)/y \equiv |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$. The numerical values of $u(x_i)/x_i$, $|\partial y/\partial x_i| \cdot (x_i/y)$, and $u_i(y)/y$, all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.
- [m] The relative standard uncertainty of $\lambda \cdot t$ is determined by the relative standard uncertainty of λ (i.e., of the half life). The relative standard uncertainty of t is negligible.
- $[n] \qquad |\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$
- [p] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.
- [q] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e. $u(x_i)/x_i = 100\%$. $|\partial y/\partial x_i| \cdot (x_i/y) = \{\text{(response per Bq of impurity)/(response per Bq of H-3)}\} \cdot \{\text{(Bq of impurity)/(Bq of H-3)}\}$. Thus $u_i(y)/y$ is the relative change in y if the impurity were present with a massic activity equal to the estimated limit of detection.

REFERENCES

- [1] International Organization for Standardization (ISO), *ISO Standards Handbook Quantities and Units*, 1993. Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.
- [2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993 (corrected and reprinted, 1995). Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.
- [3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.
- [4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814 U.S.A.
- [5] L.L. Lucas and M.P. Unterweger, *Comprehensive Review and Critical Evaluation of the Half-Life of Tritium*, J. Res. Natl. Inst. Stand. Technol. **105**, 541-549 (2000).